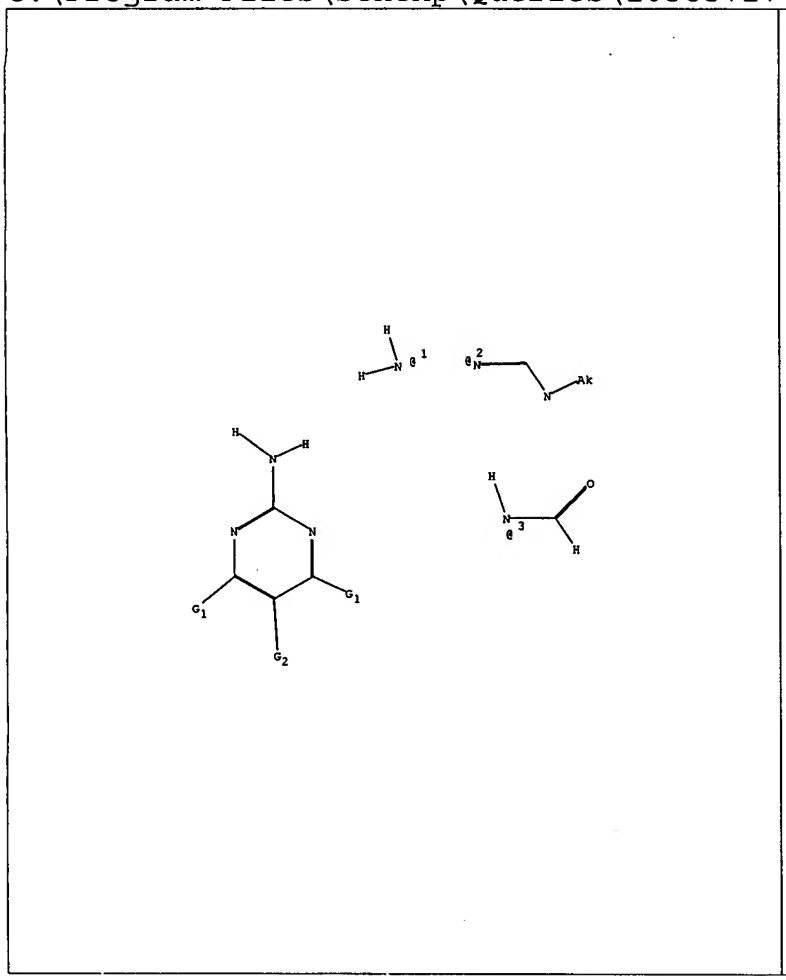
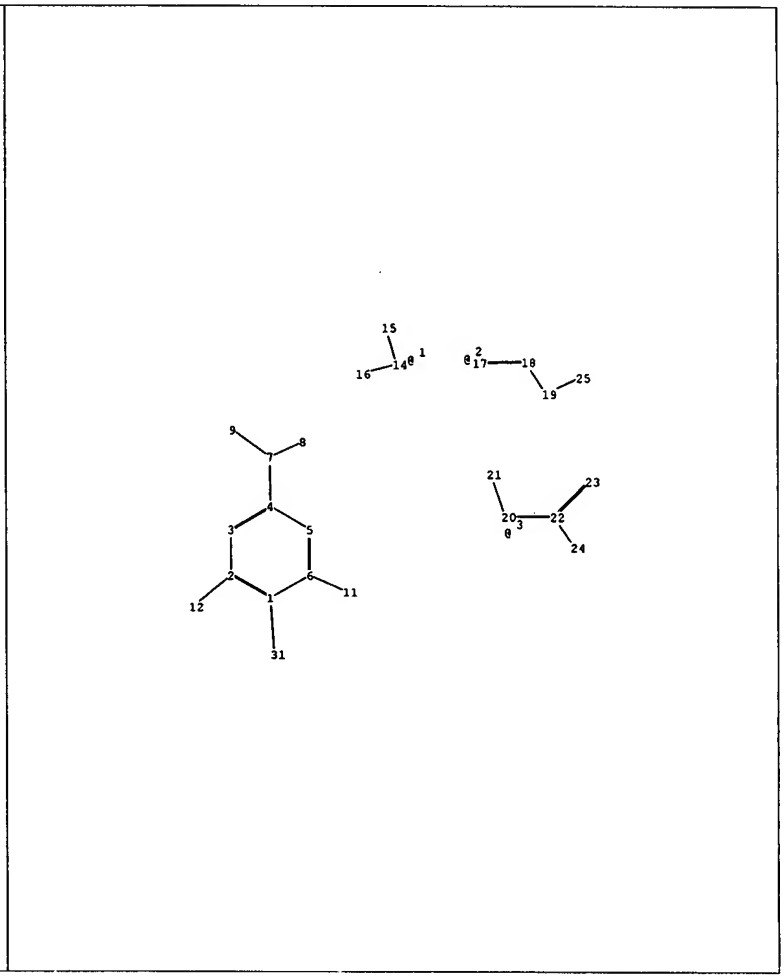
EAST Search History

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	Time Stamp
L1	7	(("5583226") or ("5663340") or ("5693800") or ("5744601") or ("6552193")).PN.	US-PGPUB; USPAT; EPO; JPO; DERWENT	OR	OFF	2007/09/30 21:27
L2	856	(544/332).CCLS.	US-PGPUB; USPAT; EPO; JPO; DERWENT	OR	OFF	2007/09/30 21:28
L3	5	Thomas.inv. and Guthner.inv. and pyrimidine	US-PGPUB; USPAT; EPO; JPO; DERWENT	OR	OFF	2007/09/30 21:30
L4	3	Karl-Heinz.inv. and neuhauser.inv. and pyrimidine	US-PGPUB; USPAT; EPO; JPO; DERWENT	OR	OFF	2007/09/30 21:30

C:\Program Files\Stnexp\Queries\10585727.str





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7 8 9 11 12 14 15 16 17 18 19 20 21 22 23 24 25 31
ring nodes :
1 2 3 4 5 6
chain bonds :
1-31 2-12 4-7 6-11 7-8 7-9 14-15 14-16 17-18 18-19 19-25 20-21
20-22 22-23 22-24
ring bonds :
1-2 1-6 2-3 3-4 4-5 5-6
exact/norm bonds :
1-31 2-12 4-7 6-11 17-18 18-19 19-25 20-22 22-23
exact bonds :
7-8 7-9 14-15 14-16 20-21 22-24
normalized bonds :
1-2 1-6 2-3 3-4 4-5 5-6
isolated ring systems :
containing 1 :

G1:OH,X

G2:[*1],[*2],[*3]

Connectivity :
25:1 E exact RC ring/chain
Match level :
1:Atom 2:Atom 3:Atom 4:Atom 5:Atom 6:Atom 7:CLASS 8:CLASS 9:CLASS
11:CLASS 12:CLASS 14:CLASS 15:CLASS 16:CLASS 17:CLASS 18:CLASS
19:CLASS

20:CLASS 21:CLASS 22:CLASS 23:CLASS 24:CLASS 25:CLASS
31:CLASS

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NEWS 4	JUL 02	CHEMCATS accession numbers revised
NEWS 5	JUL 02	CA/CAplus enhanced with utility model patents from China
NEWS 6	JUL 16	CAplus enhanced with French and German abstracts
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NEWS 9	JUL 30	USGENE now available on STN
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NEWS 11	AUG 06	BEILSTEIN updated with new compounds
NEWS 12	AUG 06	FSTA enhanced with new thesaurus edition
NEWS 13	AUG 13	CA/CAplus enhanced with additional kind codes for granted patents
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NEWS 16	AUG 27	USPATOLD now available on STN
NEWS 17	AUG 28	CAS REGISTRY enhanced with additional experimental spectral property data
NEWS 18	SEP 07	STN AnaVist, Version 2.0, now available with Derwent World Patents Index
NEWS 19	SEP 13	FORIS renamed to SOFIS
NEWS 20	SEP 13	INPADOCDB enhanced with monthly SDI frequency
NEWS 21	SEP 17	CA/CAplus enhanced with printed CA page images from 1967-1998
NEWS 22	SEP 17	CAplus coverage extended to include traditional medicine patents
NEWS 23	SEP 24	EMBASE, EMBAL, and LEMBASE reloaded with enhancements

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NEWS LOGIN	Welcome Banner and News Items
NEWS IPC8	For general information regarding STN implementation of IPC 8

Enter NEWS followed by the item number or name to see news on that specific topic.

10/585,727

* * * * * * * * * * * * * * * * STN Columbus * * * * * * * * * * * * * * * *

FILE 'HOME' ENTERED AT 20:58:14 ON 30 SEP 2007

=> file reg

COST IN U.S. DOLLARS	SINCE FILE ENTRY	TOTAL SESSION
FULL ESTIMATED COST	0.21	0.21

FILE 'REGISTRY' ENTERED AT 20:58:29 ON 30 SEP 2007
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DICTIONARY FILE UPDATES: 28 SEP 2007 HIGHEST RN 948877-55-2

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experimental property data in the original document. For information
on property searching in REGISTRY, refer to:

http://www.cas.org/support/stngen/stndoc/properties.html

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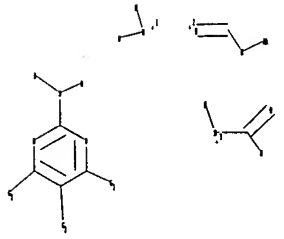

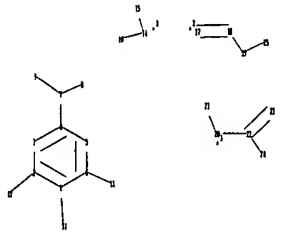

chain nodes :
7 8 9 11 12 14 15 16 17 18 19 20 21 22 23 24 25 31
ring nodes :
1 2 3 4 5 6
chain bonds :
1-31 2-12 4-7 6-11 7-8 7-9 14-15 14-16 17-18 18-19 19-25 20-21 20-22
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ring bonds :
1-2 1-6 2-3 3-4 4-5 5-6
exact/norm bonds :
1-31 2-12 4-7 6-11 17-18 18-19 19-25 20-22 22-23
exact bonds :
7-8 7-9 14-15 14-16 20-21 22-24
normalized bonds :
1-2 1-6 2-3 3-4 4-5 5-6
isolated ring systems :
containing 1 :

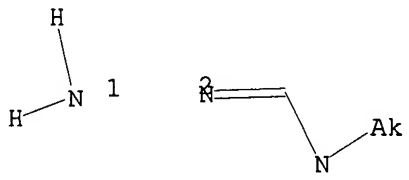
G1:OH,X

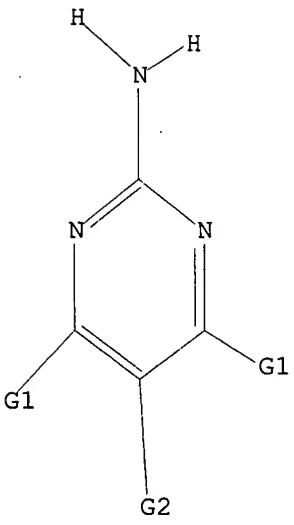
G2:[*1],[*2],[*3]

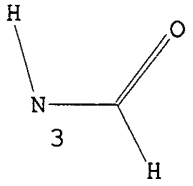
Connectivity :
25:1 E exact RC ring/chain
Match level :
1:Atom 2:Atom 3:Atom 4:Atom 5:Atom 6:Atom 7:CLASS 8:CLASS 9:CLASS 11:CLASS
12:CLASS 14:CLASS 15:CLASS 16:CLASS 17:CLASS 18:CLASS 19:CLASS 20:CLASS
21:CLASS 22:CLASS 23:CLASS 24:CLASS 25:CLASS 31:CLASS

L1 STRUCTURE UPLOADED

=> d l1
L1 HAS NO ANSWERS
L1 STR







G1 OH,X
G2 [@1],[@2],[@3]

Structure attributes must be viewed using STN Express query preparation.

=> s l1 sss sam
SAMPLE SEARCH INITIATED 20:58:58 FILE 'REGISTRY'
SAMPLE SCREEN SEARCH COMPLETED - 509 TO ITERATE

100.0% PROCESSED 509 ITERATIONS 2 ANSWERS
SEARCH TIME: 00.00.01

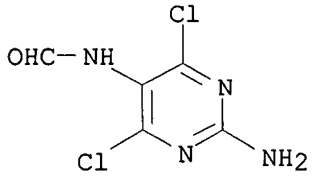
FULL FILE PROJECTIONS: ONLINE **COMPLETE**
BATCH **COMPLETE**
PROJECTED ITERATIONS: 8827 TO 11533
PROJECTED ANSWERS: 2 TO 124

L2 2 SEA SSS SAM L1

=> d scan

L2 2 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN Ethanedioic acid, monopotassium salt, compd. with N-(2-amino-4,6-dichloro-5-pyrimidinyl)formamide (1:1) (9CI)
MF C5 H4 Cl2 N4 O . C2 H2 O4 . K

CM 1



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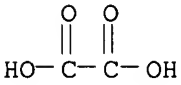
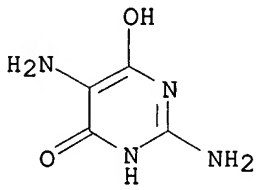


HOW MANY MORE ANSWERS DO YOU WISH TO SCAN? (1):1

10/585,727

L2 · 2 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4(1H)-Pyrimidinone, 2,5-diamino-6-hydroxy-, hydrochloride, monohydrate (9CI)
MF C4 H6 N4 O2 . x Cl H . H2 O



● x HCl

● H_2O

ALL ANSWERS HAVE BEEN SCANNED

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=> s l1 sss ful
FULL SEARCH INITIATED 20:59:15 FILE 'REGISTRY'
FULL SCREEN SEARCH COMPLETED -    10059 TO ITERATE

100.0% PROCESSED    10059 ITERATIONS                          20 ANSWERS
SEARCH TIME: 00.00.01

L3           20 SEA SSS FUL L1

=> file caplus
COST IN U.S. DOLLARS                          SINCE FILE      TOTAL
                                                   ENTRY    SESSION
FULL ESTIMATED COST                               172.10     172.31

FILE 'CAPLUS' ENTERED AT 20:59:22 ON 30 SEP 2007
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FILE LAST UPDATED: 28 Sep 2007  (20070928/ED)

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http://www.cas.org/infopolicy.html

=> s l3
L4           80 L3

=> s l3/prep
             80 L3
        4468331 PREP/RL
L5           22 L3/PREP
                   (L3 (L) PREP/RL)

=> d l5 1-22 bib abs
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L5 ANSWER 1 OF 22 CAPLUS COPYRIGHT 2007 ACS on STN
AN 2007:835093 CAPLUS
DN 147:277623
TI Method for preparing 2-amino-4,6-dichloro-5-formylaminopyrimidine
IN Jiang, Biao; Zhao, Xiaolong; Li, Yang; Li, Fan; Wang, Wanjun; Wang, Hua
PA Shanghai Intitute of Organic Chemistry, Chinese Academy of Sciences, Peop. Rep. China
SO Faming Zhuanli Shenqing Gongkai Shuomingshu, 11pp.
CODEN: CNXXEV
DT Patent
LA Chinese
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	CN 101003511	A	20070725	CN 2007-10036625	20070119
PRAI	CN 2007-10036625		20070119		

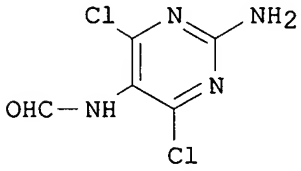
OS CASREACT 147:277623
AB The title method comprises: (1) reacting among dialkyl malonate, guanidine hydrochloride, base and organic solvent at (-20)-120°C for 1-24 h, reacting among sodium nitrite, water, organic solvent and acid containing water or not, at (-20)-120°C for 1-24 h, and reacting with reducer, and/or organic solvent at 0-120°C for 1-24 h, (2) reacting with chloridizing agent, amide compound and 2,5-diamino-4,6-dihydroxypyrimidine at a mol. ratio of (0.1-10):(0.1-10):(0.1-1) and (-20)-120°C for 1-24 h, and adding base to obtain 4,6-dichloro-5-dimethylaminomethyleneamino-2-aminopyrimidine, and (3) reacting with or without organic solvent in the presence of acid containing water or not at (-20)-120°C for 1-24 h. In step 1, the mol. ratio of dialkyl malonate, guanidine hydrochloride, base, sodium nitrite, acid and reducer is (0.1-4):(0.1-4):(0.1-16):(0.1-4):(0.1-32):(0.1-12). In step 3, the mol. ratio of 4,6-dichloro-5-dimethylaminomethyleneamino-2-aminopyrimidine and acid is (0.1-1):(0.1-20).

L5 ANSWER 2 OF 22 CAPLUS COPYRIGHT 2007 ACS on STN
AN 2006:11185 CAPLUS
DN 144:88312
TI Method for preparing 2,5-diamino-4,6-dichloropyrimidine
IN Otani, Hiroshi; Nishikawa, Junichi
PA Sumitomo Chemical Co., Ltd., Japan
SO Jpn. Kokai Tokkyo Koho, 7 pp.
CODEN: JKXXAF
DT Patent
LA Japanese
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	JP 2006001847	A	20060105	JP 2004-177545	20040615
PRAI	JP 2004-177545		20040615		

AB The title method comprises reacting N-(2-amino-4,6-dichloro-5-pyrimidinyl)formamide with acid in the presence of ammonia or an ammonium salt. The title compound is an intermediate for an antiviral nucleoside. Thus, a mixture of N-(2-amino-4,6-dichloro-5-pyrimidinyl)formamide and 35% HCl was stirred for 3 h at 20°C to 25°C; 28% ammonia water was added dropwise to said mixture with cooling; the resulting mixture was warmed to 20°C to 25°C and stirred for 1 h to give 2,5-diamino-4,6-dichloropyrimidine monohydrochloride in 82.6% yield, vs. 65% yield in a reference process.

L5 ANSWER 3 OF 22 CAPLUS COPYRIGHT 2007 ACS on STN
AN 2005:658180 CAPLUS
DN 145:145640
TI Synthesis of N-(2-amino-4,6-dichloro-5-pyrimidinyl)formamide
AU Jia, Zhi-tao; Zhang, Wei-xing; Du, Juan; Kang, Hong-jie
CS College of Material Science and Chemical Engineering, Zhejiang University, Hangzhou, 310027, Peop. Rep. China
SO Hecheng Huaxue (2005), 13(3), 270-272
CODEN: HEHUE2; ISSN: 1005-1511
PB Hecheng Huaxue Bianjibu
DT Journal
LA Chinese
OS CASREACT 145:145640
GI



I

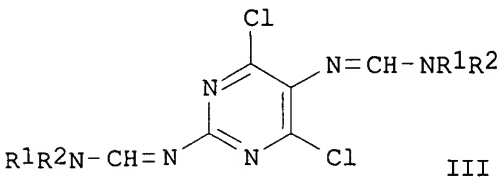
AB N-(2-amino-4,6-dichloro-5-pyrimidinyl)formamide I was synthesized from di-Et malonate and guanidine hydrochloride via cyclization, nitrosation, reduction, chlorination, condensation and acidic hydrolysis in an overall yield of 20%. The structure was characterized by 1H-NMR and IR.

L5 ANSWER 4 OF 22 CAPLUS COPYRIGHT 2007 ACS on STN
AN 2004:1037081 CAPLUS
DN 142:6554
TI Preparation of N-(2-amino-4,6-dichloropyrimidine-5-yl)formamide
IN Hayashi, Taketo; Kumazawa, Hiroharu; Kawakami, Takehiko; Nishikawa, Junichi
PA Sumitomo Chemical Company, Limited, Japan
SO PCT Int. Appl., 21 pp.
CODEN: PIXXD2
DT Patent
LA Japanese
FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI WO 2004103979	A1	20041202	WO 2004-JP7224	20040520

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW
RW: BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

PRAI JP 2003-148358 A 20030526
OS CASREACT 142:6554; MARPAT 142:6554
GI



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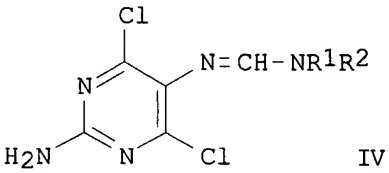

IV

AB A method for producing the title compound (I) or its salts, which comprises (a) a step of reacting 2,5-diamino-4,6-dihydroxypyrimidine (II) or its salt with R1R2NCHO (R1, R2 = alkyl, cycloalkyl, etc.) and a chlorinating agent to prepare III, (b) a step of reacting III at a pH of 3 or less, to prepare IV, and (c) a step of reacting IV at a pH of higher than 3.5 and 5 or less. Thus, I, an intermediate for synthesizing an antiviral agent, was prepared in 70.5% yield from II HCl.

RE.CNT 14 THERE ARE 14 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L5 ANSWER 5 OF 22 CAPLUS COPYRIGHT 2007 ACS on STN
AN 2004:900988 CAPLUS
DN 141:379933
TI Preparation of chloropyrimidines as intermediates for antiviral agents
IN Hayashi, Takehito; Kumasawa, Yoji; Kawakami, Takehiko
PA Sumika Fine Chemicals Co., Ltd., Japan
SO Jpn. Kokai Tokkyo Koho, 14 pp.
CODEN: JKXXAF
DT Patent
LA Japanese
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	JP 2004300101	A	20041028	JP 2003-97125	20030331
PRAI	JP 2003-97125		20030331		

OS CASREACT 141:379933; MARPAT 141:379933
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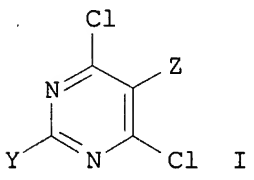


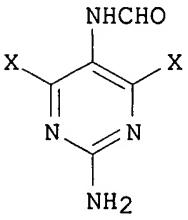
I

AB Chloropyrimidine I (Y = NH2, Z = NHCHO) or its salts, useful as intermediates for antiviral purine nucleosides, are prepared by reaction of I [Y = Z = N:CHNR1R2; R1, R2 = (un)substituted lower (cyclo)alkyl, (un)substituted aryl, (un)substituted aralkyl; R1R2 may form aliphatic heterocyclic ring] with X(CO2H)2 [X = bond, (un)substituted divalent lower hydrocarbylene]. I (Y = Z = N:CHNMe2) (prepared from 2,5-diamino-4,6-dihydroxypyrimidine) was treated with (CO2H)2 in H2O at 55° for 1 h and heated at 80° and pH 4 for 8 h to give 54.2% I (Y = NH2, Z = NHCHO).

L5 ANSWER 6 OF 22 CAPLUS COPYRIGHT 2007 ACS on STN
AN 2004:515331 CAPLUS
DN 141:54358
TI Process for the preparation of N-(2-amino-4,6-dihalopyrimidin-5-yl)formamides by formylation in acetic anhydride
IN Chemin, Eric; Cornille, Fabrice
PA Isochem, Fr.
SO Fr. Demande, 11 pp.
CODEN: FRXXBL
DT Patent
LA French
FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI FR 2849030	A1	20040625	FR 2002-16343	20021220
PRAI FR 2002-16343		20021220		

OS CASREACT 141:54358; MARPAT 141:54358
GI



I

AB The invention is related to a process for preparation of N-(2-amino-4,6-dihalogenopyrimidin-5-yl)formamides I by formylation of the corresponding 2,5-diamino-4,6-dihalogenopyrimidine or one of its salts, with formic acid in the presence of acetic anhydride [wherein X = halo, especially Cl]. The advantages include high purity product, and a simple, rapid and selective process. Thus, anhydrous HCOOH and 2,5-diamino-4,6-dichloropyrimidine reacted at 5-10° in acetic anhydride to give title compound I (X = Cl) in 68% yield and 99.2% purity.

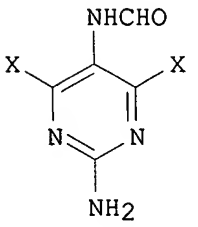
RE.CNT 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L5 ANSWER 7 OF 22 CAPLUS COPYRIGHT 2007 ACS on STN
AN 2004:205964 CAPLUS
DN 142:74474
TI Product class 12: pyrimidines
AU von Angerer, S.
CS Germany
SO Science of Synthesis (2004), 16, 379-572
CODEN: SSCYJ9
PB Georg Thieme Verlag
DT Journal; General Review
LA English
AB A review. Methods for preparing pyrimidines are reviewed including cyclization, ring transformation, aromatization and substituent modification.
RE.CNT 856 THERE ARE 856 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L5 ANSWER 8 OF 22 CAPLUS COPYRIGHT 2007 ACS on STN
AN 2000:441376 CAPLUS
DN 133:58809
TI Process for the preparation of N-(amino-4,6-dihalo-5-pyrimidinyl)formamides
IN Saikali, Elie; Brieden, Walter
PA Lonza A.-G., Switz.
SO Eur. Pat. Appl., 7 pp.
CODEN: EPXXDW
DT Patent
LA German
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 1013647	A2	20000628	EP 1999-125042	19991215
	EP 1013647	A3	20001004		
	EP 1013647	B1	20021127		
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO				
	EP 1188750	A1	20020320	EP 2001-130001	19991215
	EP 1188750	B1	20031015		
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI				
	AT 228508	T	20021215	AT 1999-125042	19991215
	PT 1013647	T	20030430	PT 1999-125042	19991215
	ES 2187112	T3	20030516	ES 1999-125042	19991215
	AT 252087	T	20031115	AT 2001-130001	19991215
	ES 2204798	T3	20040501	ES 2001-130001	19991215
	HU 9904608	A2	20000828	HU 1999-4608	19991216
	US 6271376	B1	20010807	US 1999-461244	19991216
	SK 283681	B6	20031104	SK 1999-1784	19991216
	SK 285222	B6	20060907	SK 2003-683	19991216
	JP 2000191647	A	20000711	JP 1999-359778	19991217
	JP 3543709	B2	20040721		
	CN 1265393	A	20000906	CN 1999-126244	19991217
	CZ 296832	B6	20060614	CZ 2005-24	19991217
	CZ 296753	B6	20060614	CZ 1999-4598	19991217
	NO 9906325	A	20000622	NO 1999-6325	19991220
	NO 313878	B1	20021216		
	CA 2293011	A1	20000621	CA 1999-2293011	19991221
	CA 2293011	C	20040810		
	KR 2000052540	A	20000825	KR 1999-59846	19991221
	TW 593286	B	20040621	TW 1999-88123303	19991230
	US 2001031868	A1	20011018	US 2001-867552	20010531
	US 6716981	B2	20040406		
PRAI	EP 1998-124188	A	19981221		
	EP 1999-100788	A	19990118		
	EP 1999-107161	A	19990412		
	US 1999-146106P	P	19990729		
	EP 1999-125042	A3	19991215		
	US 1999-461244	A3	19991216		

OS CASREACT 133:58809; MARPAT 133:58809
GI



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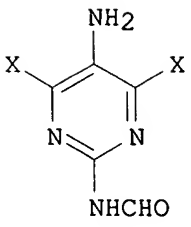


II

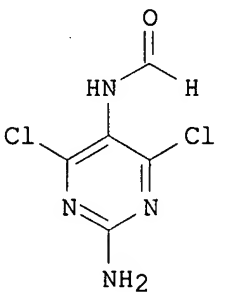
AB Title compds. I (X = halo) and II (X = halo) were prepared Thus, 0.01 mol 2,5-diamino-4,6-dichloropyrimidine and 4.55 mL water were stirred at room temperature, 14.97 mL 98% HCO2H was added, and the reaction mixture was heated at 50-55° for 3 h. After azeotropic distillation, I (X = Cl) was obtained in 90% yield.

L5 ANSWER 9 OF 22 CAPLUS COPYRIGHT 2007 ACS on STN
AN 2000:182382 CAPLUS
DN 132:334717
TI An efficient, scalable synthesis of the HIV reverse transcriptase inhibitor ziagen (1592U89)
AU Daluge, Susan M.; Martin, Michael T.; Sickles, Barry R.; Livingston, Douglas A.
CS Division of Medicinal Chemistry, Glaxo Wellcome Inc., Research Triangle Park, NC, 27709, USA
SO Nucleosides, Nucleotides & Nucleic Acids (2000), 19(1 & 2), 297-327
CODEN: NNNAFY; ISSN: 1525-7770
PB Marcel Dekker, Inc.
DT Journal
LA English
OS CASREACT 132:334717
AB Ziagen, (1S,cis)-4-[2-amino-6-(cyclopropylamino)-9H-purin-9-yl]-2-cyclopentene-1-methanol, was synthesized from (1S,4R)-azabicyclo[2.2.1]hept-5-en-3-one by efficient processes which bypass problematic steps in earlier routes. 2-Amino-4,6-dichloro-5-formamidopyrimidine is a key intermediate which makes possible an efficient construction of the purine from a chiral cyclopentenyl precursor.
RE.CNT 53 THERE ARE 53 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L5 ANSWER 10 OF 22 CAPLUS COPYRIGHT 2007 ACS on STN
AN 1996:150247 CAPLUS
DN 124:202291
TI Preparation of N-(2-amino-4,6-dichloropyrimidin-5-yl)formamide
IN Stucky, Gerhard; Imwinkelried, Rene
PA Lonza AG, Switz.
SO Eur. Pat. Appl., 13 pp.
CODEN: EPXXDW
DT Patent
LA German
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 684236	A2	19951129	EP 1995-106220	19950425
	EP 684236	A3	19960717		
	EP 684236	B1	19980624		
	R: AT, BE, CH, DE, DK, ES, FR, GB, IE, IT, LI, NL, PT, SE				
	CA 2145928	A1	19951028	CA 1995-2145928	19950330
	CA 2512305	A1	19951028	CA 1995-2512305	19950330
	JP 07300466	A	19951114	JP 1995-101499	19950425
	JP 3811966	B2	20060823		
	US 5583226	A	19961210	US 1995-428916	19950425
	EP 816344	A1	19980107	EP 1997-114001	19950425
	EP 816344	B1	20030618		
	R: AT, BE, CH, DE, DK, ES, FR, GB, IT, LI, NL, SE, PT, IE				
	AT 167672	T	19980715	AT 1995-106220	19950425
	ES 2120099	T3	19981016	ES 1995-106220	19950425
	AT 243200	T	20030715	AT 1997-114001	19950425
	PT 816344	T	20031031	PT 1997-114001	19950425
	ES 2201229	T3	20040316	ES 1997-114001	19950425
	HU 70700	A2	19951030	HU 1995-1194	19950426
	HU 219716	B	20010628		
	NO 9501594	A	19951030	NO 1995-1594	19950426
	CZ 287261	B6	20001011	CZ 1995-1067	19950426
	HU 219712	B	20010628	HU 2000-3725	19950426
	HU 219986	B	20011028	HU 2000-3724	19950426
	SK 282208	B6	20011203	SK 1995-541	19950426
	FI 9502009	A	19951028	FI 1995-2009	19950427
	FI 109119	B1	20020531		
	CN 1113237	A	19951213	CN 1995-106201	19950427
	CN 1065862	B	20010516		
	PL 190855	B1	20060228	PL 1995-308394	19950427
	TW 442474	B	20010623	TW 1995-84104525	19950506
	US 5663340	A	19970902	US 1996-693520	19960808
	US 5693800	A	19971202	US 1996-693521	19960808
	US 5744601	A	19980428	US 1997-854378	19970512
	NO 9804588	A	19951030	NO 1998-4588	19981001
	NO 306859	B1	20000103		
	CN 1259517	A	20000712	CN 1999-123455	19991102
	FI 2001001433	A	20010702	FI 2001-1433	20010702
	FI 109693	B1	20020930		
	JP 2006199707	A	20060803	JP 2006-102585	20060403
PRAI	CH 1994-1299	A	19940427		
	CA 1995-2145928	A3	19950330		
	EP 1995-106220	A3	19950425		
	JP 1995-101499	A3	19950425		
	US 1995-428916	A3	19950425		
	US 1996-693520	A3	19960808		

OS MARPAT 124:202291
GI



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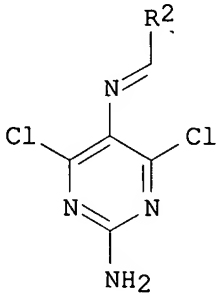


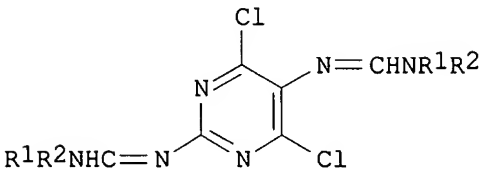
II

AB The title compound, I, is prepared in high yield and purity by the cyclization of an an aminomalonate ester R1O2CH(NH2)CO2R1 (R1 = C1-6 alkyl) or its salts with guanidine or its salts in the presence of a base (e.g., NaOMe) , forming 1,4-diamino-2,6-dihydropyridine, which is reacted with a chlorination agent (e.g., POCl3) in the presence of formamides HCOR2 [R2 = (un)substituted NH2 or heterocyclic ring] (e.g., DMF) to yield a dichloropyrimidine, II, which is subsequently reacted with an alkanoic acid (e.g., AcOH, etc.).

L5 ANSWER 11 OF 22 CAPLUS COPYRIGHT 2007 ACS on STN
AN 1995:994188 CAPLUS
DN 124:56577
TI Preparation of chloropyrimidine intermediates for 9-substituted-2-aminopurines.
IN Daluge, Susan Many; Martin, Michael Tolar; Fugett, Michelle Joanne Ferry
PA Wellcome Foundation, Ltd., UK
SO PCT Int. Appl., 35 pp.
CODEN: PIXXD2
DT Patent
LA English
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 9521161	A1	19950810	WO 1995-GB225	19950203
	W: AM, AT, AU, BB, BG, BR, BY, CA, CH, CN, CZ, DE, DK, EE, ES, FI, GB, GE, HU, JP, KE, KG, KP, KR, KZ, LK, LR, LT, LU, LV, MD, MG, MN, MW, MX, NL, NO, NZ, PL, PT, RO, RU, SD, SE, SI, SK, TJ, TT, UA, US				
	RW: KE, MW, SD, SZ, AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG				
	CA 2182105	A1	19950810	CA 1995-2182105	19950203
	CA 2182105	C	20060725		
	AU 9515438	A	19950821	AU 1995-15438	19950203
	AU 690203	B2	19980423		
	ZA 9500884	A	19960805	ZA 1995-884	19950203
	EP 741710	A1	19961113	EP 1995-907107	19950203
	EP 741710	B1	20000510		
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LI, LU, MC, NL, PT, SE				
	CN 1139924	A	19970108	CN 1995-191478	19950203
	CN 1105109	B	20030409		
	HU 75300	A2	19970528	HU 1996-2114	19950203
	HU 223096	B1	20040329		
	JP 09508412	T	19970826	JP 1995-520467	19950203
	JP 3670012	B2	20050713		
	BR 9506667	A	19970916	BR 1995-6667	19950203
	RU 2140913	C1	19991110	RU 1996-118435	19950203
	AT 192742	T	20000515	AT 1995-907107	19950203
	IL 112539	A	20000831	IL 1995-112539	19950203
	PT 741710	T	20000929	PT 1995-907107	19950203
	ES 2148486	T3	20001016	ES 1995-907107	19950203
	PL 183885	B1	20020731	PL 1995-315713	19950203
	IL 129935	A	20041215	IL 1995-129935	19950203
	TW 390877	B	20000521	TW 1995-84102412	19950314
	US 6448403	B1	20020910	US 1996-682743	19960731
	FI 9603070	A	19960802	FI 1996-3070	19960802
	FI 112477	B1	20031215		
	NO 9603239	A	19961002	NO 1996-3239	19960802
	NO 310819	B1	20010903		
	US 5917041	A	19990629	US 1997-957043	19971024
	US 5917042	A	19990629	US 1997-957605	19971024
	US 6087501	A	20000711	US 1997-957603	19971024
	US 6555687	B1	20030429	US 1997-957606	19971024
	US 6552193	B1	20030422	US 1999-419416	19991015
	GR 3033850	T3	20001031	GR 2000-401551	20000630
	CN 1388123	A	20030101	CN 2002-102344	20020117
	US 2002173649	A1	20021121	US 2002-184482	20020627
	US 6870053	B2	20050322		
	US 2003187263	A1	20031002	US 2003-389815	20030318
PRAI	GB 1994-2161	A	19940204		
	IL 1995-112539	A3	19950203		
	WO 1995-GB225	W	19950203		

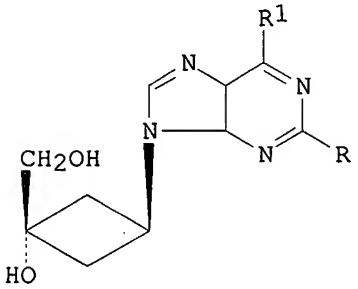
US 1996-682743 A3 19960731
US 1997-957045 A1 19971024
US 1997-957603 A1 19971024
OS MARPAT 124:56577
GI



I

AB Title compds. [I; R1, R2 = alkyl, cycloalkyl, (substituted) aryl], were prepared Thus, 2,5-diamino-4,6-dihydroxypyrimidine hemisulfate and Vilsmeier reagent were refluxed in CH2Cl2 to give 81% I (R1 = R2 = Me), which was converted to (1S,4R)-4-[2-amino-6-(cyclopropylamino)-9H-purin-9-yl]-2-cyclopentene-1-methanol in several steps.

L5 ANSWER 12 OF 22 CAPLUS COPYRIGHT 2007 ACS on STN
AN 1991:536621 CAPLUS
DN 115:136621
TI Synthesis of carbocyclic oxetanocin analogs as potential anti-HIV agents. Part 3
AU Boumchita, Hassane; Legraverend, Michel; Guilhem, Jean; Bisagni, Emile
CS Inst. Curie, Cent. Univ., Orsay, 91405, Fr.
SO Heterocycles (1991), 32(5), 867-71
CODEN: HTCYAM; ISSN: 0385-5414
DT Journal
LA English
OS CASREACT 115:136621
GI



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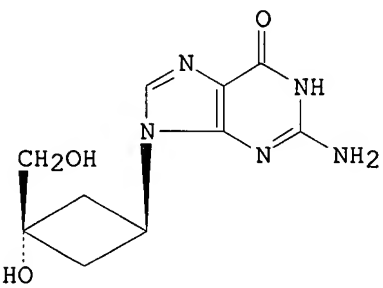


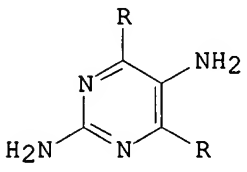
II

AB Two new carbocyclic oxetanocin analogs I (R = H, R1 = NH2) and II were prepared from 1-amino-3-methylenecyclobutane. The results of biol. testing against HIV-1 in vitro are presented. The crystal structures of intermediates I (R = H, NH2, R1 = Cl) were determined

L5 ANSWER 13 OF 22 CAPLUS COPYRIGHT 2007 ACS on STN
AN 1991:185542 CAPLUS
DN 114:185542
TI Process for preparing 2,5-diamino-4,6-dichloropyrimidine
IN Hanson, John Christopher
PA Beecham Group PLC, UK
SO PCT Int. Appl., 14 pp.
CODEN: PIXXD2
DT Patent
LA English
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 9101310	A1	19910207	WO 1990-GB1109	19900719
	W: AU, CA, JP, KR, US				
	RW: AT, BE, CH, DE, DK, ES, FR, GB, IT, LU, NL, SE				
	CA 2063827	A1	19910122	CA 1990-2063827	19900719
	AU 9060364	A	19910222	AU 1990-60364	19900719
	AU 634564	B2	19930225		
	EP 483204	A1	19920506	EP 1990-910799	19900719
	EP 483204	B1	19950524		
	R: AT, BE, CH, DE, DK, ES, FR, GB, IT, LI, LU, NL, SE				
	JP 04506802	T	19921126	JP 1990-510336	19900719
	ES 2074577	T3	19950916	ES 1990-910799	19900719
	US 5216161	A	19930601	US 1992-820890	19920116
PRAI	GB 1989-16698	A	19890721		
	WO 1990-GB1109	A	19900719		

OS CASREACT 114:185542
GI

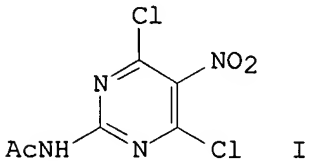


I

AB Chloride I (R = Cl) was prepared by treating I (R = OH) with POCl3 in a quaternary ammonium chloride or a tertiary amine hydrochloride. Et3N+Me Cl-, Et4N+Cl-, and N-ethyl-N-methylpiperidinium chloride were used.

L5 ANSWER 14 OF 22 CAPLUS COPYRIGHT 2007 ACS on STN
AN 1991:6424 CAPLUS
DN 114:6424
TI A new route to 2,5-diamino-4,6-dichloropyrimidine, a key precursor of 9-substituted guanines
AU Legraverend, Michel; Boumchita, Hassane; Bisagni, Emile
CS Inst. Curie, Cent. Univ., Orsay, F-91405, Fr.
SO Synthesis (1990), (7), 587-9
CODEN: SYNTBF; ISSN: 0039-7881
DT Journal
LA English
OS CASREACT 114:6424
AB An improved synthesis of 2,5-diamino-4,6-dihydroxypyrimidine (I) is reported. The direct chlorination of I provides the shortest (2 step) synthesis of 2,5-diamino-4,6-dichloropyrimidine (II) reported to data. The procedure described here affords an easy approach to II a key intermediate to various 9-substituted guanines.

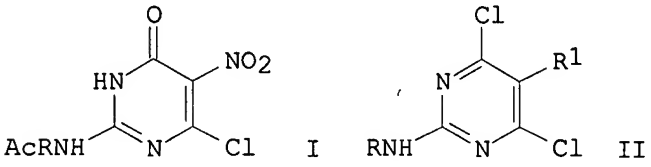
L5 ANSWER 15 OF 22 CAPLUS COPYRIGHT 2007 ACS on STN
AN 1980:215381 CAPLUS
Correction of: 1978:615347
DN 92:215381
Correction of: 89:215347
TI Preparation of 2,5-diamino-4,6-dichloropyrimidine via N-(4,6-dichloro-5-nitropyrimidin-2-yl)acetamide. The preparation of 2-aminopyrimidine intermediates
AU Temple, Carroll, Jr.; Smith, Buford H.; Montgomery, John A.
CS South. Res. Inst., Kettering-Meyer Lab., Birmingham, AL, 35205, USA
SO Nucleic Acid Chem. (1978), Volume 1, 47-52. Editor(s): Townsend, Leroy B.; Tipson, R. Stuart. Publisher: Wiley, New York, N. Y.
CODEN: 39GCA6
DT Conference
LA English
GI



I

AB Nitration of 2-amino-6-chloro-4-pyrimidinone gave the 5-nitro derivative, which was acetylated and chlorinated with POCl3 to give I. Reduction of I and deacetylation gave 2,5-diamino-4,6-dichloropyrimidine.

L5 ANSWER 16 OF 22 CAPLUS COPYRIGHT 2007 ACS on STN
AN 1978:615347 CAPLUS
DN 89:215347
TI Preparation of 2,5-diamino-4,6-dichloropyrimidine via N-(4,6-dichloro-5-nitropyrimidin-2-yl)acetamide: The preparation of 2-aminopyrimidine intermediates
AU Temple, Carroll, Jr.; Smith, Buford H.; Montgomery, John A.
CS Kettering-Meyer Lab., Southern Res. Inst., Birmingham, AL, USA
SO Nucleic Acid Chem. (1978), Volume 1, 47-52. Editor(s): Townsend, Leroy B.; Tipson, R. Stuart. Publisher: Wiley, New York, N. Y.
CODEN: 39GCA6
DT Conference
LA English
GI



AB Nitration of 2-amino-6-chloro-4-pyrimidinone followed by acetylation gave pyrimidinone I, which was treated with POCl3 to give II (R = Ac, R1 = NO2). The latter was reduced to give II (R = Ac, R1 = NH2), which was hydrolyzed to give II (R = H, R1 = NH2).

L5 ANSWER 17 OF 22 CAPLUS COPYRIGHT 2007 ACS on STN
AN 1975:578974 CAPLUS
DN 83:178974
TI Preparation of 2,5-diamino-4,6-dichloropyrimidine
AU Temple, Carroll, Jr.; Smith, Buford H.; Montgomery, John A.
CS Kettering-Meyer Lab., South. Res. Inst., Birmingham, AL, USA
SO Journal of Organic Chemistry (1975), 40(21), 3141-2
CODEN: JOCEAH; ISSN: 0022-3263
DT Journal
LA English
OS CASREACT 83:178974
GI For diagram(s), see printed CA Issue.
AB The pyrimidine I (R = R2 = H, R1 = Cl) was nitrated and treated with Ac2O to give I (R = NO2, R1 = Cl, R2 = Ac), which was treated with POCl3 and the product reduced to give the title compound (II). I (R = NH2, R1 = OH, R2 = H) and Ac2O gave III.

L5 ANSWER 18 OF 22 CAPLUS COPYRIGHT 2007 ACS on STN
AN 1970:100634 CAPLUS
DN 72:100634
TI Conversion of ureidomalonates and 5-carbalkoxyhydantoins to 5-ureido-4,6-pyrimidinediones
AU Perini, Florian R.; Tieckelmann, Howard
CS Dep. of Chem., State Univ. of New York, Buffalo, NY, USA
SO Journal of Organic Chemistry (1970), 35(3), 812-16
CODEN: JOCEAH; ISSN: 0022-3263
DT Journal
LA English
AB Both ureidomalonates and 5-carbethoxyhydantoins were readily condensed with guanidine to give the same products, 2-amino-5-(N'-substituted-ureido)-4,6-pyrimidinediones (I) in good yield. Acid-catalyzed cyclization of I produced 8-hydroxyguanines. Chlorination and acylation of the ureidopyrimi-dinediones were studied. Thiourea condensed with the ureido-malonates, but urea did not.

L5 ANSWER 19 OF 22 CAPLUS COPYRIGHT 2007 ACS on STN
AN 1958:104328 CAPLUS
DN 52:104328
OREF 52:18426h-i,18427a-i,18428a-b
TI Potential purine antagonists. XI. Synthesis of some 9-aryl(alkyl)-2,6-disubstituted purines
AU Koppel, Henry C.; Robins, Roland K.
CS Arizona State Coll., Tempe
SO Journal of the American Chemical Society (1958), 80, 2751-5
CODEN: JACSAT; ISSN: 0002-7863
DT Journal
LA Unavailable
OS CASREACT 52:104328
AB cf. C.A. 52, 13741h. NaNO2 (40 g.) added to 100 g. barbituric acid in 1 l. H2O at 70-80°, allowed to stand 10 min., treated with stirring with 200 g. Na2S2O4 in portions below 90°, cooled to room temperature, and filtered yielded 92 g. 5-amino-2,4,6-trihydroxypyrimidine (uracil) (I). I (70 g.) in 1500 cc. N NaOH treated at 60° with stirring dropwise with 66 g. PhNCS during about 1.5 h., stirred 2 h. at 60°, acidified with glacial AcOH, cooled, and filtered yielded 95 g. N-(2,4,6-trihydroxy-5-pyrimidyl)-N'-phenylthiourea, plates, m. above 300°. 2,6-Dihydroxy-9-phenyl-8-purinethiol (50 g.) in 500 cc. N NaOH refluxed 3 h. with 150 g. wet Raney Ni, filtered, cooled to 4°, and filtered again, the filtrate refluxed again 3 h. with 150 g. fresh Raney Ni and processed as before, the combined filter residues dissolved in boiling H2O, and the solution treated with C and acidified with concentrated HCl gave 20 g. 2,6-dihydroxy-9-phenylpurine (II), plates, m. above 300°. II (8 g.) and 24 g. P2S5 ground together, diluted with 500 cc. dry pyridine, refluxed 3 h., the excess pyridine removed in vacuo, the residue diluted with 500 cc. iced H2O, the solution kept at room temperature, refluxed 2 h., acidified with HCl, cooled, and the crude product (4.5 g.) repptd. twice from hot dilute aqueous KOH gave 2-hydroxy-9-phenyl-6-purinethiol-H2O, light yellow needles, m. above 300°; it lost 1 mol H2O at 180°. II (20 g.), 500 cc. POCl3, and 100 g. PCl5 refluxed 40 h., the excess POCl3 removed in vacuo, the residue poured with stirring onto crushed ice, the solution extracted with six 1-l. portions Et2O, and the extract worked up gave 12 g. 2,6-dichloro-9-phenylpurine (III), pale yellow needles, m. 244-6° (EtOAc); the insol. residue (3.0 g.) from the Et2O extraction boiled in N NaOH gave 1.2 g. 2-chloro-6-hydroxy-9-phenylpurine (IV). III (3 g.) refluxed 3 h. in N NaOH, the solution treated with C, filtered, chilled, the precipitate filtered off, washed, dissolved in boiling H2O, and the solution acidified with glacial AcOH while hot gave 1.9 g. IV, needles, m. 280-1° (EtOH). III (5 g.) added to 200 cc. absolute MeOH containing 10 g. CS-(NH2)2, refluxed 6 h., and cooled yielded 3 g. 9-phenyl-2,-6-purinedithiol, light green needles, m. above 300° (90% EtOH). III (5 g.) in 100 cc. EtOH heated on the steam bath with 12 cc. PrNH2 to solution and then an addnl. 3 h. and cooled gave 4.0 g. 2-chloro-6-propylamino-9-phenylpurine, needles, m. 121-2° (decomposition) (80% EtOH). III (4 g.) added to 75 cc. absolute EtOH containing 1.9 g. Ph(CH2)2NH2, heated 1.5 h. on the steam bath, treated with C, filtered, cooled, and treated 20 min. with a stream of dry HCl gave 5.4 g. 2-chloro-6(2-phenylethylamino)-9-phenylpurine-HCl (V.HCl), m. 172-4° (absolute EtOH). III (5 g.) in 70 cc. H2O heated 8 h. on the steam bath with 20 cc. 40% aqueous Me2NH, cooled, and filtered gave 3.5 g. 6-(Me2N) analog of V, needles, m. 168-9° (EtOH). N-(2,4,6-Trihydroxy-5-pyrimidyl)-N'-(p-chlorophenyl)thiourea (40 g.) refluxed 5 h. in 650 cc. concentrated HCl, diluted to 1 l. with H2O, and filtered immediately gave 23 g. 2,6-dihydroxy-9-(p-chlorophenyl)-8-purinethiol (VI), light yellow, m. above 300° (aqueous AcOH). VI (30 g.) in 500

cc. N NaOH refluxed 3 h. with 90 g. wet Raney Ni, filtered, cooled to 10°, and filtered again yielded 9.0 g. Na salt of the p-Cl deriv, of II. 2,6-Dihydroxy-9-methyl-8-purinethiol (VII) (10 g.) treated similarly with Raney Ni and the resulting Na salt acidified with glacial AcOH gave 4.8 g. 2,6-dihydroxy-9-methylpurine (VIII), m. above 300°. The 9-Et homolog of VII (17.0 g.) gave similarly 6 g. 9-Et homolog of VIII. N-(2,4,6-Trihydroxy-5-pyrimidyl)-N'isobutylthiourea (25 g.) refluxed 5 h. in 250 cc. concentrated HCl, diluted to 500 cc. with H2O, and filtered immediately yielded 16 g. 9-iso-Bu analog (IX) of VI. IX (10 g.) in 200 cc. N NaOH refluxed 3 h. with 30 g. Raney Ni yielded 5.0 g. 9-iso-Bu homolog of VIII. 2-Amino-4,6-dihydroxypyrimidine (100 g.) in 800 cc. 0.5N NaOH treated at 60° with 40 g. NaNO2 and then with concentrated HCl, filtered, the residue washed with a little H2O, suspended in 1 l. H2O at 20°, treated carefully with 25 g. Na2S2O4, boiled 5 min., and filtered hot, and the deposit from the filtrate recrystd. from H2O gave 38 g. 2,5-diamino-4,6-dihydroxypyrimidine (X). X (25 g.) in 400 cc. N NaOH treated at 60-70° with 13 g. MeNCS, stirred 4 h., acidified with glacial AcOH, kept 6 h. at room temperature, and filtered yielded 25 g. N-(2-amino-4,6-dihydroxy-5-pyrimidyl)-N'-methylurea (XI); it became highly colored in air. Crude XI (25 g.) and 250 cc. concentrated HCl refluxed 5 h., diluted to 450 cc. with H2O, and filtered immediately gave 14 g. crude 2-amino-6-hydroxy-9-methyl-8-purinethiol, which refluxed successively in the usual manner with two 42-g. portions wet Raney Ni in 250 cc. N NaOH yielded 7.5 g. 2-amino-6-hydroxy-9-methylpurine (XII), m. above 300° (aqueous HCONMe2). X (23 g.) treated in the usual manner with 20 g. iso-BuNCS and the resulting N-(2-amino-4,6-dihydroxy-5-pyrimidyl)-N'-isobutylurea cyclized in HCl gave 12 g. crude product which desulfurized in the usual manner with two 40-g. portions wet Raney Ni in 250 cc. N NaOH gave 5.1 g. 9-iso-Bu homolog of XII. X (25 g.) treated with 17 g. EtNCS, the resulting product cyclized with concentrated HCl, and the 2-amino-6-hydroxy-9-ethyl-8-purinethiol (14 g.) desulfurized with Raney Ni in the usual manner gave 6.0 g. 9-Et homolog of XII. XII (8 g.) and 32 g. P2S5 in 500 cc. dry pyridine refluxed 8 h., the pyridine removed in vacuo, the residue treated with 500 cc. iced H2O, the solution heated 3 h. on the steam bath and chilled overnight, and the crude deposit (5.0 g.) precipitated twice from hot, dilute aqueous NaOH with AcOH gave 2-amino-9-methyl-6-purinethiol, light yellow, m. above 300°. I (71 g.) in 1.5 l. N NaOH treated at 60-70° dropwise with stirring with 75 g. p-ClC6H4NCO during about 1.5 h., stirred 2 h. at 60-70°, cooled, acidified with glacial AcOH filtered, the residue washed with a little H2O and refluxed 6 h. with 1 l. concentrated HCl, diluted with H2O to 1500 cc., and the precipitate washed with H2O and dried gave 70 g. 9-(p-chlorophenyl)uric acid (XIII), needles, m. above 300° (AcOH). I (54 g.) and 50 g. o-ClC6H4NCO gave similarly 49.0 g. o-isomer of XIII, needles, m. above 300° (aqueous AcOH). The UV absorption maximum of the substituted purines reported are tabulated.

L5 ANSWER 20 OF 22 CAPLUS COPYRIGHT 2007 ACS on STN
AN 1955:56753 CAPLUS
DN 49:56753
OREF 49:10972a-e
TI Pyrimidines. IV. The synthesis of several new chlorosubstituted pyrimidines
AU Robins, Roland K.; Dille, K. L.; Christensen, Bert E.
CS Oregon State Coll., Corvallis
SO Journal of Organic Chemistry (1954), 19, 930-3
CODEN: JOCEAH; ISSN: 0022-3263
DT Journal
LA Unavailable
OS CASREACT 49:56753
AB cf. C.A. 43, 1424b. Addition of 65 g. Et2NPh to 25 g. 5-nitrobarbituric acid and 100 cc. POCl3 at 25-30° during 30 min., warming at 45-50° 25 min., addition to ice and Et2O extraction gave 20.2% 2,4,6,5-Cl3(O2N)R [R = the pyrimidine nucleus in this abstract] (I), m. 57-8° (from heptane). Slow addition of 1.0 g. I in dry Et2O to Et2O saturated with dry NH3 at 0°, keeping at 0° 1 hr., evaporation of solvent and extraction of the residue with C6H6 gave 0.3 g. 2,6,4,5-Cl2(H2N)O2NR (II), m. 162-4° (cf. m.p. 152° reported by Bittleri and Erlenmeyer, C.A. 46, 513a; II prepared by method of B. and E. also m. 162-4°). 2,4,6,5-Cl(H2N)2O2NR (3.0 g.) by addition of 4 g. I in EtOH to EtOH saturated with NH3, m. above 300° (decomposition). Hydrogenation of 3.0 g. I over Raney Ni in 95% EtOH at 10 lbs. H pressure/sq. in. 2 hrs. gave 2.8 g. 2,4,6,5-Cl3(H2N)R (III), m. 116-17°. Slow bubbling of NH3 into a solution of 4.0 g. III, and 230 cc. 3N NH4OH at 90-5°, refluxing 30 min. and cooling gave 2.1 g. 2,5,4,6-(H2N)2Cl2R, m. 260-1°. 4,6,5-Cl2(H2N)R, 2.1 g. from hydrogenation of 3.0 g. nitro analog, m. 147-8°; this compound does not react with hot 15% NH4OH in 30 min. Gentle refluxing of 0.63 g. 2,6,4,5-Cl2(H2N)2R and 5 cc. 98% HCO2H 15 min., concentrating to dryness and crystallizing from hot NH4OH (pH 8) gave 41% 2,6,4,5-Cl2(H2N)HCONHR, m. 216-17°. 2,4,5-Cl(H2N)2R sulfate was prepared in 45% yield by solution of the free base in 5% H2SO4 and cooling.

L5 ANSWER 21 OF 22 CAPLUS COPYRIGHT 2007 ACS on STN
AN 1953:66026 CAPLUS
DN 47:66026
OREF 47:11205d-i,11206a-i,11207a-b
TI Condensed pyrimidine systems. X. Some 1,3-oxazolo[5,4-d]pyrimidines
AU Falco, Elvira A.; Elion, Gertrude B.; Burgi, Elizabeth; Hitchings, George H.
CS Wellcome Research Labs, New York, NY
SO Journal of the American Chemical Society (1952), 74, 4897-4902
CODEN: JACSAT; ISSN: 0002-7863
DT Journal
LA Unavailable
GI For diagram(s), see printed CA Issue.
AB cf. C.A. 47, 8662c. A series of 1,3-oxazolo[5,4-d]pyrimidines has been prepared from 5-acylamino-4-pyrimidinols with POCl3. 6-Amino-5-acylamino-4-pyrimidinols gave with POCl3 2 products, the alkali-soluble major product being a purine, while the alkali-insol. fraction present in highly variable, usually minor, proportion was an oxazolopyrimidine (I); with carefully dried amide and freshly distilled POCl3 the amount of I was reduced to a trace, whereas a maximum yield of I was obtained when the amide was treated with POCl3 containing 0.5 mol. H2O/mol. The conversion of representative I to purines by heating with amines could be demonstrated. The I could be differentiated from certain oxazinopyrimidines by their UV absorption spectra. The following 5-acylamino-4-pyrimidinols, (II), (X, Y, and R given) were prepared from the corresponding 5-aminopyrimidines and acyl halides in aqueous solution according to Wilson (C.A. 43, 652a): Me, Me, Ph, m. 282°; Me, Me, p-MeC6H4, m. 278-9°; Me, Me, p-ClC6H4, m. 310-15° (decomposition); Me, Me, p-O2NC6H4, m. 320° (decomposition); H, NH2, m-O2NC6H4, m. 305° (decomposition); and H, NH2, p-ClC6H4 (III), m. 340-50° (decomposition). BzNHCH(CHO)CO2Et (IV) (22 g.) was converted to the Na derivative and let stand 72 h. with 5.0 g. HC(:NH)NH2.HCl in 100 cc. EtOH at room temperature to yield 1.9 g. II with H, H, Ph, m. 249-50°. Crude Na derivative (48 g.) of IV let stand with 4.6 g. MeC(:NH)NH2.HCl and 2.7 g. KOH in 150 cc. H2O at room temperature gave 2.45 g. II with Me, H, Ph, m. 294-5° (from EtOH). 5-Amino-2,6-dimethyl-4-pyrimidinol (2 g.) was heated 0.5 h. with 30 cc. 98% HCO2H, evaporated to dryness, and the residue taken up in 20 cc. H2O and neutralized with dilute NH4OH to yield II with Me, Me, H, needles, m. 245-8° (from 95% EtOH). 5-Amino-2-dimethylamino-6-methyl-4-pyrimidinol (1 g.) refluxed 1 h. with 20 cc. Ac2O gave 800 mg. II with Me2N, Me, Me, m. 225-7°. The II heated 2-3 h. with 10 cc. POCl3/g. II, the excess POCl3 removed in vacuo, the sirupy residue poured on ice, and the mixture made alkaline, gave the corresponding 1,3-oxazolo[5,4-d]pyrimidines (I), which were filtered off (2-Ph derivs.) or extracted with Et2O. The following I [X, Y, R, % yield, m.p. (sublimation temperature in parentheses) given]: Me, Me, Ph, 60, 108-9° (from H2O); Me, H, Ph, 68, 122° (from H2O); Me, Me, p-MeC6H4, 38, 176-7° (from H2O); Me, Me, H, 10, 118-19° (80-90°); Me, Me, p-ClC6H4, 73, 196-7° (from 95% EtOH); Me, Me, p-O2NC6H4, 10, 224-5° (from MeOH); Me, Me, p-H2NC6H4, 50, 193 (decomposition) (from 95% EtOH); NMe2, Me, Me, 5, 83-4° (40°) [the reactants were refluxed 15 h., λmaximum 255 mμ (ε 18300), 325 mμ (3280), λmin. 230 (5950), 285 (1340) at pH 1, and λmaximum 260 (16800), 320 (5560), and λmin. 285 (1430) at pH 11]; Cl, H, Ph (V), 62, 165-7° (110-50°) (the reactants were heated 36 h.); NH2, NH2, p-ClC6H4 (VI), 316-18° (decomposition) (from EtOAc), λmaximum 310 and λmin. 255 at pH 1, λmaximum 242 and 315, and λmin. 265 at pH 11; NH2, NH2, m-O2NC6H4, 50, 291-2° (decomposition) (from EtOAc); NH2, NH2, p-BrC6H4, 10, 320-1° (from EtOAc); NH2, NH2, o-BrC6H4, 4, 247-8° (from EtOAc); H, H, Ph, 52, 113-16° (sublimed); and H, NH2, m-O2NC6H4, 11, 263-6° (from EtOAc). III (14 g.) refluxed 3 h. with 140 g.

com. POCl3, the excess POCl3 removed in vacuo, the residue poured on ice, the mixture adjusted with 2N NaOH to pH 10, filtered, and the filtrate neutralized with AcOH gave 8-(p-chlorophenyl)-6-chloropurine, crystals from 95% EtOH, λmaximum 242 (12700) and 305 (30000), and λmin. 255 (6150) at pH 1, λmaximum 240 (19900) and 330 (18800), and λmin. 260 (3850) at pH 11; the residue from the alkaline filtrate, recrystd. from EtOAc, gave 1.4 g. (10%) 7-amino-2-(p-chlorophenyl)-1,3-oxazolo[5,4-d]pyrimidine (VII), pale yellow plates, m. above 320°, λmaximum 295 and λmin. 250 at pH 1, and λmaximum 242 and 295, and λmin. 270 at pH 11. V (160 mg.) heated 16 h. with alc. NH3 (saturated at 0°) at 140° gave 5-amino-2-phenyl-1,3-oxazolo[5,4-d]pyrimidine, m. 285-7° (from EtOH). VI (175 mg.) refluxed 6 h. with 60 cc. 6N HCl and the mixture filtered from some p-ClC6H4CO2H and neutralized gave 2-amino-5-(p-chlorobenzamido)-4,6-pyrimidinediol (VIII), did not melt at 320°, λmaximum 255 (20800) at pH 1 and 240 (16000) and 253 (13750) at pH 11. 2,5-Diamino-4,6-pyrimidinediol (IX).HCl (150 mg.) treated with 0.11 cc. p-ClC6H4COCl by the method of Wilson (loc. cit.) and the product washed with 10 cc. Et2O, dissolved in alkali, and precipitated with dilute AcOH gave VIII.

V (1 g.) heated at 160° 16 h. with 50 alc. NH3 and the product dissolved in 3% alc. HCl and precipitated with Et2O gave VIII.2HCl.2H2O; the filtrate from the crude reaction product evaporated to dryness and the residue recrystd. from a small amount of H2O gave 120 mg. 2-amino-8-phenylpurine, faintly pink needles, m. 265-8°, λmaximum 260 (23200), 335 (12400) and λmin. 235 (13800), 285 (7900) at pH 1, and λmaximum 240 (17200), 330 (18800), and λmin. 275 (6550), also obtained by heating 5-benzamido-2,4-diaminopyrimidine, (750 mg.) 0.5 h. at 205-10°. VII and 10% alc. MeNH2 heated in a sealed tube 16 h. at 160° gave 6-amino-8-(p-chlorophenyl)-9-methyl-β-purine, pale pink needles, λmaximum 238 (15500), 297 (23100) and λmin. 255 (7350) at pH 1, and λmaximum 243 (20800) 313 (20400) and λmin. 270 (9300). V (60 mg.) and 100 cc. alc. NH3 heated 70 h. at 160° in a sealed tube gave 6-amino-8-(p-chlorophenyl)purine, also obtained by heating 5-(p-chlorobenzamido)-4,6-diaminopyrimidine 1 h. at 200°; recrystd. from 2N HCl it gave the HCl salt. VI heated 96 h. with alc. NH3 at 160° gave 8-(p-chlorophenyl)-2,6-diaminopurine.

2-Amino-5-(chloroacetamido)-6-methyl-4-pyrimidinol (X) was refluxed 1.5 h. with 20 cc. POCl3 and 1.3 cc. H2O, the excess POCl3 removed in vacuo, the residue poured on ice, the mixture neutralized with NH4OH to pH 8.5, extracted 3 times with 100-cc. portions of Et2O and 3 times with 100-cc. portions of C6H6, and the combined exts. were dried and evaporated to give 5-amino-2-(chloromethyl)-7-methyl-1-3-oxazolo[5,4-d]pyrimidine (XI), m. 238-9° (decomposition) (from C6H6). XI let stand 3 h. with 2.5N H2SO4 at room temperature caused hydrolytic cleavage of the oxazole ring, as evidenced by the UV absorption spectrum; XI boiled with 2N NaOH gave 2,4-diamino-6-methyl-4-pyrimidinol. The 2-dimethylamino analog (XII) of X. (750 mg.), m. 258°, heated 15 h. with 50 cc. POCl3 and the product sublimed at 120° and 0.03 mm. gave a compound C9H12Cl2N4O, colorless needles, m. 168-70°. A similar run with 10 g. added PCl5 gave a small amount of a compound C9H11Cl3N4. XIII (350 mg.) heated 1.5 h. with 4 cc. POCl3 and 0.65 cc. H2O gave 150 mg. of a crystalline product, m. 107-8°, subliming in needles, which contained apparently as the major component 2-chloromethyl-5-dimethylamino-7-methyl-1,3-oxazolo[5,4-d]pyrimidine. The UV absorption maximum and min. and ε values (in parentheses) at pH 1, and also at pH 11 (in brackets) are given for the following compds.: XI, maximum 245 (25800), 310 (8410), min. 275 (3180), [maximum 250 (14900), 300 (11200), min. 275 (4480)]; IX (free base), maximum 255 (6150), min. 233 (4250), [maximum 242 (7270), 295 (4920), min. 270 (3350)]; 6-amino-8-phenylpurine, maximum 238 (15500), 297 (23100), min. 255 (7350),

[maximum 243 (20800), 313 (20400), min. 270 (9300)]; 2-amino-4-methyl-5H-p-oxazino[2,3-d]pyrimidin-6-ol, maximum 255 (8650), 310 (3060), min. 240 (4500), 290 (2520), [maximum 243 (5950), 295 (5200), min. 262 (4500)]; and 2-dimethylamino-4-methyl-5H-p-oxazino[2,3-d]-pyrimidine, maximum 235 (10900), 285 (11500), 325 (3170), min. 250 (8500), 295 (2180), [maximum 280 (11300), min. 245 (3560)].

L5 ANSWER 22 OF 22 CAPLUS COPYRIGHT 2007 ACS on STN
AN 1949:17543 CAPLUS
DN 43:17543
OREF 43:3425h-i,3426a-d
TI Isomeric dihydroxanthopterins
AU Hitchings, George H.; Elion, Gertrude B.
SO Journal of the American Chemical Society (1949), 71, 467-73
CODEN: JACSAT; ISSN: 0002-7863
DT Journal
LA Unavailable
GI For diagram(s), see printed CA Issue.
AB 2,4,5-Triamino-6-hydroxypyrimidine (I) (8 g.) and 24 g. ClCH2CO2H, heated 1 hr. on the water bath, give 95% of the 5-chloroacetamido compound (IA), with 1 mol. H2O; absorption spectra of I and IA given at pH 1 and 11. IA (11.9 g.) and 8.4 g. NaHCO3 in 350 ml. H2O, heated 2 hrs. at 95°, give 68.5% β-dihydroxanthopterin (II), soluble in about 5000 parts boiling H2O, readily soluble in warm 2.5 N HCl, from which the mono-HCl salt seps. as needles; the sulfate, with 4 mols. H2O, loses 3 mols. at 150° (2 hrs.); picrate, dark yellow, m. 265° (decomposition). The ultraviolet absorption of II and the α-isomer (III) (O'Dell, et al., C.A. 41, 4155f) are given at pH 1, 3, 7, and 11. II is not oxidized by O with Pt catalyst or in alkali; neutral KMnO4 gives a glycol (?). II (0.4 g.) and 0.63 g. Ba(OH)2.8H2O in 15 ml. H2O, heated 6 hrs. on the water bath, give 0.2 g. 2,5-diamino-6-hydroxy-4-(carboxymethylamino)pyrimidine, with 1.5 mols. H2O, does not m. up to 350°; ultraviolet absorption spectra at pH 1 and 11 given; warm 0.2 N HCl gives II; MeOH-HCl gives the Me ester, which is converted to II by heating to 200°, by solution in alkali, or simply by standing in H2O. III seps. with 1 mol. H2O (0.5 mol. lost at 130°, the other half at 150°). III is not cleaved by Ba(OH)2; the sulfate seps. with 1 mol. H2O; picrate, pinkish orange, does not m. at 370°. III is oxidized to xanthopterin by Ag2O and by alkaline KMnO4; with more than 2 equivs. KMnO4 it gives a glycol (?). The structure of III is unknown. 2,5-Diamino-4,6-dihydroxypyrimidine (IV) (5 g.) and 13 g. (ClCH2CO)2O, heated 1 hr. on the water bath, give 80% of the 5-chloroacetamido compound (V), with 1 mol. H2O; absorption curves are given for IV, V, and 8-methylguanine.

10/585,727

=> log y

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